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पेट्रोलियम और इसके उत्पादों की परीक्षण पद्धतियां

[पी : 140]

पारा पिकनोमीटर पद्धति द्वारा पेट्रोलियम कोक का प्रत्यक्ष घनत्व ज्ञात करना

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 140]

DETERMINATION OF APPARENT DENSITY OF PETROLEUM COKE BY MERCURY PYKNOMETER METHOD

UDC 665·777·4 : 531·756

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1992

Price Group 2

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 140]

DETERMINATION OF APPARENT DENSITY OF PETROLEUM COKE BY MERCURY PYKNOMETER METHOD

1 SCOPE

1.1 This method covers the determination of the apparent density of coke by the mercury pyknometer method.

2 SIGNIFICANCE AND USE

2.1 Mercury density analysis gives information of the amount of pitch needed for mixing with coke for anode production. Mercury in this analysis is analogous to the pitch in anode production. It assists in determining the percentage of the pitch in the paste. If the apparent density is decreasing it is an indication that the porosity of the coke has increased, requiring more pitch to be added for good binding.

2.2 The apparent density of coke in mercury under 1 bar (1 bar = 10^5 Pa) pressure is linked with the geometric density of green and baked anodes.

2.3 Open porosity value corresponding to accessible pores of diameter less than 15 micron may be calculated knowing the mercury apparent density and helium apparent density of coke, using the same size fractions and thus the amount of pitch to be added for anode production.

3 OUTLINE OF THE METHOD

A known amount of coke is weighed and placed in a calibrated pyknometer. The pyknometer is evacuated and then mercury is introduced. The mass of the mercury necessary to fill the pyknometer up to the volume is determined and then apparent density is calculated. After knowing the two apparent densities the open porosity of petroleum coke may be calculated by the following equation:

$$V = 1\,000\left[\frac{1}{\text{AD (M)}} - \frac{1}{\text{AD(H)}}\right]$$

AD (M) = Apparent Density in Hg (g/cm^3)

AD (H) = Apparent Density in Helium (g/cm^3)

V = Open porosity, mm^3/g

4 REAGENTS

4.1 Mercury, A. R

4.2 Ethanol (95 percent) or Methylated Spirit

5 APPARATUS

5.1 Duplex dilatometer, filling and macropore determination device; (*see* Fig. 1)

NOTE — The test procedure described here is specific to Duplex dilatometer filling and macropore determination device from Carlo Erba Strumentazione, P. O. Box 434 20100, Milano (Italy). Similar instruments by which identical results may be obtained are also suitable.

5.2 Pyknometer,50 ml glass, fabricated as per drawing (*see* Fig 2).

5.3 Rotary High Vacuum Pump, capable of producing a vacuum upto 0·001 mm of mercury.

5.4 Vacuastat, capable of measuring vacuum upto 0·001 mm of mercury.

5.5 Jaw Crusher, laboratory type; manganese steel jaw plates.

5.6 Drying Oven, maintained at 105 – 110°C;

5.7 Sieves, Tyler No. 10 (1·65 mm) and Tyler No. 20 (0·85 mm) or equivalent.

5.8 Filtration Apparatus

5.9 Analytical Balance

5.10 Desiccator, glass

6 SAFETY PRECAUTIONS

6.1 Goggles and hand gloves shall be worn when handling mercury and working under a vacuum.

6.2 It is imperative that spilt mercury is removed immediately, due to the high toxicity of its vapour. A small aspirator bottle with a capillary tube and connected to a pump may be used for sucking up mercury droplets. Material spilt into crevices may be made non-volatile by putting zinc dust into the cracks to form an amalgam.

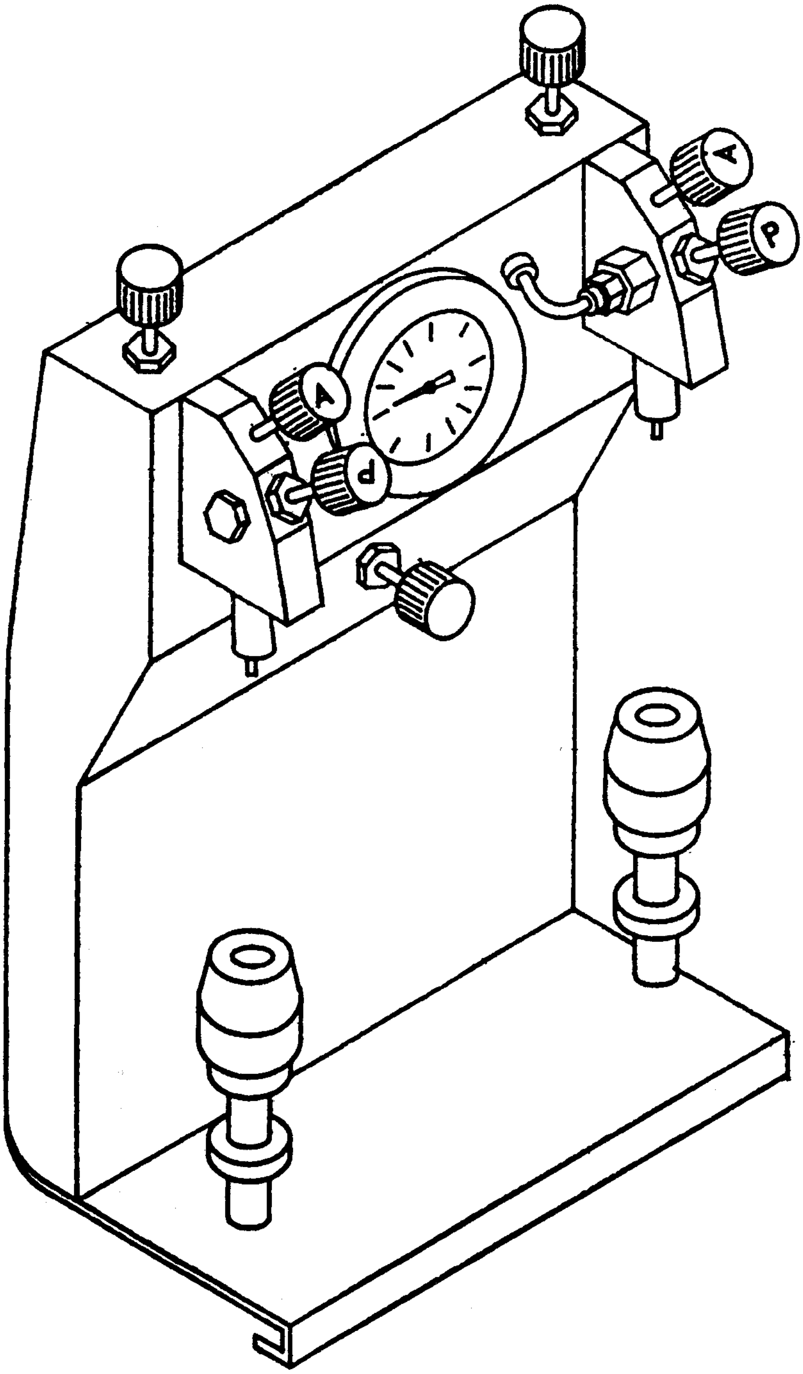
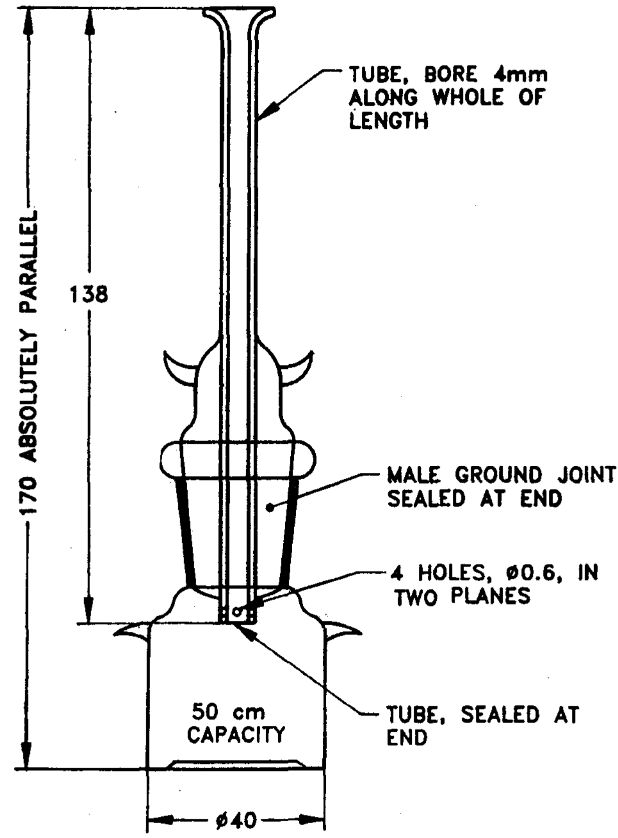


FIG. 1 CARLO ERBA APPARATUS


TUBE, BORE 4mm ALONG WHOLE OF LENGTH
170 ABSOLUTELY PARALLEL
138
MALE GROUND JOINT, SEALED AT END
4 HOLES, ø0.6, IN TWO PLANES
50 cm CAPACITY
TUBE, SEALED AT END
ø40

All dimensions in millimetres.

FIG. 2 PYKNOMETER FOR DETERMINATION OF APPARENT DENSITY TO MERCURY

7 SAMPLE PREPARATION

7.1 The representative sample is passed through a Jaw crusher rated to give grains smaller than 1·65 mm.

7.2 The crushed sample is shifted and the −1·6 mm to + 0·85 mm (10/20) fraction is retained for analysis.

7.3 The fraction is dried for one hour in an oven maintained at 105 — 110°C and then closed in a desiccator.

8 PROCEDURE

8.1 Calibration

Pyknometer shall be calibrated before use. Because of difference in technique, each analyst shall calibrate the pyknometer themselves and not use a calibration mass obtained by another analyst. Calibration shall be checked at least every six months.

8.1.1 A reference mark is made on the capillary at a height "*h*" measured from the mid-point of the pyknometer bulb such that h (cm) = 85 — H where H is the mean atmospheric pressure expressed as cm of mercury.

NOTE — The value to be employed is the average atmospheric pressure for the locality. No allowance be made for diurnal or seasonal fluctuations.

8.1.2 Place the empty pyknometer in the Carlo Erba apparatus ensuring a tight seal is made and all valves are closed.

8.1.3 Turn on the vacuum pump.

8.1.4 Open valve '*P*' and wait for 10 minutes.

8.1.5 Open valve '*M*' Mercury will flow into the pyknometer. Tap the pyknometer gently.

8.1.6 Close valve '*M*' when mercury reaches the reference mark.

8.1.7 Close valve '*P*'.

8.1.8 Slowly open valve '*A*' to increase the pressure to atmospheric pressure.

8.1.9 When pressure has equalized with atmosphere, remove the pyknometer from Carlo Erba Apparatus.

8.1.10 Record the mass of the mercury filled in pyknometer.

8.2 Sample Analysis

8.2.1 Weigh 10 ± 0·001 g of the prepared coke sample.

8.2.2 Place in the pyknometer. Attach the greased pyknometer top.

8.2.3 Place the pyknometer with the sample on the Carlo Erba apparatus. Ensure a tight seal has formed and all valves are closed.

8.2.4 Switch on the vacuum pump and slowly open valve '*P*' and evacuate till the vacuum gauge indicates 0·01 mm of Hg (time will depend on degree of calcination of the sample). Maintain this vacuum for at least 30 minutes.

8.2.5 While maintaining the vacuum (valve '*P*' open) open valve '*M*'. Mercury will flow on to the pyknometer. Tap it gently to ensure the complete transfer of mercury to the vicinity of the mark.

8.2.6 Close valve 'M' when the mercury reaches slightly above the reference mark.

8.2.7 Close valve '*P*'.

8.2.8 Slowly open valve '*A*' to increase the pressure to atmosphere pressure.

8.2.9 When pressure has equalized with atmosphere, remove the pyknometer from Carlo Erba apparatus. Adjust the height of mercury in the capillary to the mark by withdrawing the excess mercury, care being taken not to jar the pyknometer.

8.2.10 Record the mass of the pyknometer containing the coke and mercury.

8.2.11 All experimental procedures shall be carried out at 25 ± 1°C.

8.3 Cleaning of the Pyknometer

8.3.1 The pyknometer is cleaned by pouring the mercury and the sample through the filtration apparatus, separating the mercury from the coke.

8.3.2 Clean inside of pyknometer with tissues and alcohol.

8.3.3 Return mercury into the reservoir on Carlo Erba apparatus.

9 CALCULATION

$$\text{Apparent Density, g/ml} = \frac{\text{mass of the sample in g}}{\text{volume of mercury displaced by the sample}}$$

The mass of coke is approximately 10·0 g. The volume of the sample is determined by using the volume of mercury excluded from the pyknometer.

$$\text{Apparent Density (g/ml)} = \frac{M \times 13{\cdot}534}{M_1 - M_2 + M}$$

where

M = mass of the sample in g;

M_1 = mass of the pyknometer in g, filled to the mark with mercury;

M_2 = mass of the pyknometer in g, plus contents after the analysis; and

13·534 = Density of the mercury in g/ml at 27°C.

10 REPORTING

Report the apparent densities to two decimal places.

11 PRECISION

Results of duplicate tests shall not differ by more than the following amounts:

Repeatability	*Reproducibility*
0·01 from the mean result	0·02 from the mean result

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